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(54) **Cleaning articles, substrates therefor, and method of substrate manufacture**

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Produits de nettoyage, substrat à cet effet, et procédé de sa fabrication

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Description

[0001] This invention relates to cleaning articles, substrates therefor, and a process for the manufacture of such substrates. The invention has particular application to substrates which are to be used to produce so-called wet wipes, i.e. small sheets, moistened with a liquid having cleansing or other relevant properties. Such wipes are sold, for example, in the form of baby wipes, adult incontinent wipes, and facial/skin cleaning wipes. It is to be understood, however, that the invention is also applicable to other forms of product used for cleaning areas of the human person, for example moist toilet paper and dry wipes, and is also applicable to wipes intended for cleaning other surfaces, for example kitchen and bathroom surfaces, and surfaces which require cleaning in industry, for example surfaces of machinery or of automobiles. As will be apparent from the ensuing description, some, though not all, of the advantages afforded by the present invention and embodiments thereof are relevant to cleaning such inanimate surfaces.

[0002] For simplicity, the description below concentrates on wet wipes for cleaning the human skin, but what is said must be understood in the light of the foregoing comments about the wider applicability of the invention.

[0003] Wet wipes commonly comprise a substrate of a non-woven fibrous material, wetted by a suitable liquid. The non-woven substrate material is formed to have a high strength. Some limitations may be imposed on the strength of the substrate if it is desired that its density should be low enough to permit the presence of pores in which a significant amount of the wetting liquid can be held. However, this is not much of a limitation, and it may be no limitation at all, since all or most of the wetting liquid can be held by virtue of the strongly hydrophilic surfaces of the fibres, rather than by the capillary action of any open pores between the fibres. This is true, for example in the case of cellulose/pulp air laid tissues. Typically, a known wet wipe substrate will have a breaking tensile strength of about 50 or 60 N in MD and about 8N in CD, where MD and CD refer to machine direction (i.e. the direction of travel of the substrate through the machine which is producing it) and cross direction (a direction in the plane of the substrate and at 90° to machine direction).

[0004] An alternative method of defining strength, and one which may be more appropriate for the purpose of comparing these known substrates with substrates according to the invention described below, is the work, typically expressed in Nm (Newton-metres) required to stretch the substrate to breaking point. This is referred to below as toughness. A test for measuring toughness is described in more detail below.

[0005] One method of manufacturing non-woven substrates involves a process known as hydroentangling (see EP-A-0 166 060). In this, a web of fibres, for example a carded web, travels beneath at least one array of orifices, and preferably a plurality of from successive arrays, from which jets of water are emitted. Each array extends transversely with respect to the path of travel of the web, and provides a large number of closely spaced jets. These jets act like sharp needles, and entangle the fibres to form a substrate. This entanglement holds the fibres in a coherent substrate, without the need for adhesives or thermal bonding. In current equipment, the orifices typically have a diameter of from about 80 to about 180 micrometers, preferably about 90 to 150 micrometers, and there may typically be from about 800 to 1700 nozzles per metre of orifice array. The water is supplied to the orifice arrays at a pressure which generally increases stepwise from the first array, where it may be as little as 30 bar, to the last array, where it may be as much as about 250 bar. This stepwise increase is provided to allow for the fact that the fibres are progressively more and more difficult to move as entanglement proceeds. The total energy supplied to the web by the liquid jets from all the orifice arrays combined is 0.5 to 1.0 kWh/kg of fibre material.

[0006] It has now surprisingly been found that wet wipes can be produced which have superior properties to known wet wipes, both as regards as their cleaning ability and as regards the softness which the user perceives them to have, if the substrate is one which is significantly less strong than the substrates of known wet wipes, and provided certain other conditions are fulfilled. In the case of substrates made using a hydroentangling process, the lower strength can be achieved by reducing total energy input of liquid jets.

[0007] It should be mentioned that although hydroentangling is preferred as the method of producing the substrate, other methods known per se in the art can alternatively be used. For example, the water can be replaced by another liquid, or by a gas, for example air or steam. Alternatively, the fluid "needles" can be replaced by mechanical needles, in a process known as needlepunching. In this barbed needles, e.g. of steel, are punched through the web, hooking tufts of fibres across it and thereby bonding it in the needlepunched areas. The needles enter and leave the web while it is trapped between two plates, the web being pulled through the apparatus by draw rolls.

[0008] Another method which might be used is thermal bonding, in which the fibres are of thermoplastic material, or have an outer layer of thermoplastic material, and are bonded together in discrete spots by heat. This bonding can be achieved using a drum which has at least one heating element in the interior thereof, and which has an exterior surface carrying an embossed pattern with which the fibrous layer is pressed into contact. However, a substrate produced in this way is unlikely to be as soft as one produced by hydrogentangling, or needlepunching, and may have a substantially proportion of completely loose fibres.

[0009] Yet another method involves wet-laying a mixture of fibres and chemical binder, somewhat in the manner employed, for example, in paper making.

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[0010] Needlepunching, thermal bonding, and wet-laying are all well known per se and will therefore not be described in detail below. Hydroentangling is also well known per se, but is described further below, because it is the preferred method and for the purpose of mentioning a number of modifications to the hydroentangling procedure as it is usually practised.

5 [0011] Thus, according to the present invention there is provided a non-woven fibrous substrate for use in a cleaning article, wherein the substrate has a major surface for rubbing on a surface to be cleaned, a low strength in at least one direction, and comprises at least a proportion of long fibres which are capable of protruding from the said major surface as a result of said rubbing whilst remaining attached to the substrate.

10 [0012] The substrate, as made, has a toughness of less than 0.6Nm in at least one direction, preferably not more than 0.5 Nm. The toughness is preferably less than 1.2 Nm in each of two mutually perpendicular directions. The tensile strength is preferably not more than 45 N. After rubbing is applied, in a manner described below the toughness is preferably reduced by at least 15%, and is preferably less than 0.5 Nm, in at least one direction, and less than 0.6 in each of two mutually perpendicular directions. the tensile strength after rubbing is preferably not more than 10N in at least one direction, and not more than 30N in each of two mutually perpendicular directions.

15 [0013] The invention further provides a wet wipe comprising a substrate as aforesaid and a cleaning liquid:

20 [0014] The invention also provides a method of producing a non-woven, fibrous substrate for use in a cleaning article, and having a major surface for rubbing on a surface to be cleaned, wherein a fibrous layer comprising fibres of which at least a proportion are long fibres, is subjected to hydroentangling by means of jets of liquid which apply to the fibrous layer a force sufficient to produce a coherent substrate but low enough for at least some of said long fibres to be capable of protruding from said major surface as a result of said rubbing, whilst remaining attached to the substrate.

[0015] A substrate according to the invention must thus possess two significant characteristics identified as (a) and (b) below, and should desirably possess two others, identified as (c) and (d). These are:

25 (a) Low strength

[0016] This is necessary in order to allow significant quantities of surface fibres to become separated from the surface plane of the substrate when the substrate is subjected to the friction which results from rubbing on the skin or other surface to be cleaned. The low strength may be in MD or CD or both.

30 (b) The presence of relatively long fibres

35 [0017] At least a certain proportion of the fibres, preferably at least 20%, more preferably at least 40%, still more preferably at least 60%, and yet more preferably at least 80%, are sufficiently long that even though they extend from the surface plane of the substrate as a result of the friction produced by rubbing, they nevertheless remain attached to the body of the substrate. In one preferred form, substantially all the fibres are long fibres. Such fibres can either remain attached at one end, with the other end extending from the substrate surface, or they can remain attached at both ends, but have a central portion extending from the substrate surface. The fibres referred to herein as "long" have a length of at least 2cm, normally from 2 to 6cm, and more preferably at least 3cm, normally from 3 to 5cm.

40 (c) The ability of loosened fibre portions to remain away from the substrate surface

45 [0018] The point here is that the desired effect is unlikely to be achieved if the loosened fibre portions have a tendency to remain against the substrate surface. One way in which this tendency can be avoided is if at least a proportion of the fibres concerned has a relatively high flexural stiffness, even when the substrate is wetted by a cleaning liquid, if present, and so will tend to protrude from the substrate surface. To give this stiffness, such fibres should preferably have a tensile strength, when dry, of at least 2.5cN, more preferably at least 5cN, and still more preferably of the order of 10cN. However, they should not be so stiff, at least where the product is to be used on the skin, as to give the product an unacceptably abrasive feel, and the tensile strength should therefore preferably not exceed 30cN, and more preferably should not exceed 20cN.

50 [0019] Where the substrate is to be used for an article, such as a wet wipe, containing a cleaning liquid which is either an aqueous solution or an emulsion in which the continuous phase is aqueous, instead of, or in addition to, the fibres having at least a given flexural stiffness, the desired effect may be achieved if those fibres are hydrophobic, and thus resist the absorption of the cleaning liquid which the wet wipe contains. Fibre materials which have suitably hydrophobic properties include polyolefins such as polypropylene and polyethylene, polyamides such as nylon, and polyethylene terephthalate.

55 [0020] If the liquid carried by the wipe or cleaning article is oil-based, for example in the form of an emulsion in which the oil forms the continuous phase, the fibres could be caused to protrude from the surface of the substrate by their being hydrophilic, and suitable fibre materials for this purpose include viscose fibres and cotton fibres.

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[0021] Yet another possibility is for the cleaning liquid to be a silicone emulsion, in which case the fibres must be such as will continue to protrude from the substrate in the presence of such an emulsion.

(d) Texturing

[0022] This is very desirable, for reasons which will be apparent from what is said below.

[0023] In the accompanying drawings:

Figure 1 is a diagrammatic perspective view of an example of an apparatus which can be used to produce a substrate according to the present invention, using hydroentangling;

Figure 2 is a set of graphs showing the strength of various substrates, some according to the invention and some not; and

Figures 2 and 3 are photographs with a 1.75:1 magnification, showing a substrate according to the invention before and after rubbing;

Figures 6 and 5 are cross section, magnified x25, through a substrate according to the invention and a comparative substrate;

Figures 6a to 6f are graphs generated by an Instron machine test (described further below) of various substrates; and

Figure 7 shows a Lissajous curve, referred to below in connection with a rubbing test referred to below.

[0024] The apparatus 10 shown in Figure 1 comprises a continuous belt 12, on the upper run of which the hydro-entangling process takes place. As viewed in Figure 1, the upper run travels in a rightward direction and preferably does so at a speed of 25-75 m/min, more preferably 40-60 m/min. The belt is apertured, as described in more detail below. A layer of fibres 14, such as a nonwoven batt or other initial fibrous layer is fed on to the belt, as indicated diagrammatically by arrow 16. The initial fibrous layer may consist of any suitable web, mat or batt of loose fibres, disposed in random relationship with one another, or in any degree of alignment, such as might be produced by carding or the like. The initial fibrous layer may be made by any desired technique, such as by carding, random laydown, air or slurry deposition or the like.

[0025] The initial fibrous layer then passes beneath a plurality of orifice arrays 18. Each array extends transversely across the line of travel of the belt 12 and fibrous layer 14. In the drawing, five such arrays 18 are shown, but it must be emphasised that the number of arrays could be more or less than this, say from 2 to 15. There might be only a single nozzle array, but a plurality is preferable. Each array 18 has a perforated plate on the underside thereof in which is formed at least one row of orifices. Typically, there is a single row or a pair of parallel rows, and where there is a pair of rows the orifices in one row may be staggered with respect to the orifices in the other row. The row, or each row, as the case may be, runs parallel to the length of the array 18. The orifices used in one preferred form of the present invention have a diameter of from 100-120 micrometers, and are arranged, for example, at about 0.6mm centres.

[0026] Water is fed to the arrays via a high pressure line 20 and individual pressure regulators 22, one per array. By this means the pressure can differ from array to array. Thus, as already mentioned, the pressure may increase stepwise from one array to the next, as considered from the upstream end to the downstream end of the belt. The sum total of the pressure applied to individual arrays is preferably from 40-1100 bars, more preferably from 40-200 bars. The water flow rate through each array is preferably from 2-5 m³/hr for each metre length of the array. The flow velocity of each water jet is from 40 to 110 m/sec, more preferably from 40 to 100 m/sec, and the energy flow per water jet is preferably from 300-14000 bars.m/sec, more preferably at least 800 bars.m/sec. The total power input to the web from all the arrays combined, is preferably from 1.0-120 kW/m of web width, and is more preferably not more than 100 kW/m. The total energy input to the web is from 0.005 to 0.8 kWh/kg of material, and preferably less than 0.5 kWh/kg. More preferably it is in a range of from 0.1 to less than 0.5 kWh/kg. Still more preferably the upper limit of this range is 0.4 or 0.3, and the lower limit of the range may be 0.2.

[0027] Vacuum boxes 24, one for each array 18, are provided beneath the belt, for the purpose of taking away the water after it has passed through the belt.

[0028] As already mentioned the belt is in the form of a wire grid, preferably a rectangular, and, more preferably, square, grid, defining a corresponding array of apertures. For example, a grid may be used in which there are from 12 to 30 apertures per cm², preferably from 20 to 30 apertures per cm². It must be understood, however, that finer or coarser grids may be used, and that the apertures may be arranged in ways which do not constitute square or rectangular grids.

[0029] As explained above, the water is used to supply an amount of energy which is low compared to that conventionally used, though sufficient to effect hydroentangling. One effect of this is that the substrate thus formed does not have openings extending through it even at the locations where, during its formation, it is immediately above the wire crossing points in the belt, at least as regards a substantial proportion of those locations. Preferably, not more than

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50% of the locations form open apertures, and more preferably not more than 30%. Generally, at least a few percent of the locations will provide open apertures, say at least 5%. Except where the apertures are open, the substrate consists of areas of reduced thickness formed above the wire crossing points and ridges elsewhere. The absence of large numbers of openings in the substrate is an advantage where, for example, it is to be used as the substrate for a wet wipe. If many openings were present these could permit dirt and other undesirable materials to pass through the substrate from the surface being cleaned to, for example, the hand of the user.

[0030] The existence of the above mentioned ridges in the substrate has at least two significant advantages. One is that it increases the caliper of the substrate material without a proportionate increase in the amount of material required to make it. The other is that the ridges form initiation areas where friction produced during rubbing can start the partial break-up of the surface of the substrate, with the attendant desirable effects already described.

[0031] The amount, and nature, of the entangling produced by the water jets depends, inter alia on the belt speed and the direction of the jets. Regarding the direction of belt travel (MD) as the y direction, the direction transverse thereto in the plane of the belt (CD) as the x direction, and the direction perpendicular to the plane of the belt as the z direction, the greater the difference between the component of belt velocity in the x or y direction, and the component of jet velocity in the same direction, the greater will be the entangling effect in that direction.

[0032] Thus, when the belt is travelling at a high speed in the y direction, and the jets are aimed directly along the z axis, as is conventional in a hydroentangling apparatus, there is a substantial amount of entangling in both directions, but much more in the y direction than in the x direction. Correspondingly, the resulting strength of the substrate in the x direction is much less than in the y direction. The faster the belt speed, the higher the proportion of the energy which goes into the y direction entangling, and the lower the proportion which goes into the x direction.

[0033] The respective strength in these two directions can be altered by angling the jets upstream (increasing the y direction entangling), downstream (decreasing the y direction entangling), laterally (increasing the x direction entangling), or in some combination thereof. For example, the jets can be angled upstream or downstream by up to 45° with respect to the normal to the belt, and/or laterally by up to 45°. In addition, some jets could point in different directions to other jets, either within a given array or from one array to another. Further, some or all of the orifices could be shaped to give a swirling motion to the jets issuing therefrom.

[0034] In the foregoing description, the surface on which hydroentangling takes place has been referred to as a belt. It is to be understood, however, this is only by way of example, and the surface could be any suitable travelling surface, whether travelling linearly or, in the case of a drum for example, circularly.

[0035] Also, the apparatus shown in Figure 1 has water jets only on one surface of the substrate. It may be desirable to effect hydroentangling from both surfaces. In that case, the web of Figure 1 can be passed through a second, oppositely disposed assembly of orifices arrays and vacuum boxes. Alternatively, the initial web can pass through an assembly in which orifice arrays on one side of the web alternate with orifice arrays on the other side.

[0036] Further general information about hydroentangling can be found, for example, in US-A-2862251 (Kalwaites), US-A-3025585 (Griswold), US-A-3485706 (Evans), US-A-5204158 (Phillips et al), US-A-5320900 (Oathout) and EP-A-0418493 (Fiberwet), the contents of which are incorporated herein by reference. Hydroentangling equipment suitable for use in carrying out the present invention is obtainable from ICBT Perfojet, Z.A. Pre-Millet, 38330 Mont Bonnot, France.

[0037] Various fibre compositions can be used to produce the substrate, though preferably the composition should include sufficient fibres which have the desired ability to protrude from the surface of the substrate. Examples of fibre compositions which can be used include mixtures of a hydrophilic fibre material (e.g. viscose, cotton or flax) and a hydrophobic fibre material (e.g. polyethylene terephthalate or polypropylene), which may be present in any desired proportions, or purely hydrophilic or purely hydrophobic materials. Preferably there is from 30-70% of a hydrophilic material and 70%-30% of a hydrophobic material. All these percentages are by weight. Two particularly preferred compositions are 50% viscose/50% PP and 50% viscose/50% PET. One advantage provided by having such a large amount of viscose fibres is that their surfaces are ridged, having ridges of the order of 10 micrometers high, and their ridges provide additional friction to help initiate partial break-up of the substrate.

[0038] The substrate preferably has a basis weight of at least 30 gm². Where the substrate is to be used for a wet wipe it is unlikely that a basis weight of more than 150 gm² will be required, though in theory higher basis weights could be employed. Indeed, given that the invention is concerned with what is, in essence, a surface effect, it can be seen that it is not unreasonable that, provided the substrate is not too thin, its thickness should be, at least largely, irrelevant to the invention. For practical purposes, however, at least in the field of ordinary wet wipes, the basis weight is preferably in the range of 30-130 gm² more preferably 30-70 gm², and still more preferably 55 to 65 gm². A product having a basis weight of 55 to 65 gm², when made according to the present invention by a hydroentangling process, and with a textured surface, typically has a caliper of about 0.8mm, including the texturing. The texturing, as already mentioned can be achieved by carrying out the hydroentangling on an apertured surface. If the overall caliper is less than about 0.4 mm it may be difficult to incorporate a textured surface, and such a surface is advantageous, for the reasons already given. Preferably, the caliper of the substrate is from 0.4mm to 2.0mm, more preferably from 0.4 to

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0.95mm. The bulk density of the substrate is preferably not more than 0.1g/cm³, preferably not more than 0.9g/cm³, and still preferably not more than 0.8g/cm³. In determining the density, the volume of the substrate is calculated using the caliper of the substrate including the texturing.

[0039] As already indicated, a substrate produced according to the present invention results in superior cleaning properties to those obtained by substrates according to the prior art. The principle reason for this is believed to be the fact that the fibres which protrude from the substrate surface as the cleaning process proceeds provide additional cleaning surfaces. However, the actual cleaning mechanism may involve other effects in addition to, or instead of, the one just described, and no reliance is to be placed on the particular theoretical explanation just given.

[0040] As well as the improved cleaning effect, there is also an increase in substrate softness. This is believed to result from two mechanisms. Firstly, the low strength of the material results in a material-imposed limitation on the level of friction which can be achieved between the substrate and the surface of which it is being rubbed, for example the skin. When the frictional force begins to exceed that limit, individual fibres partially tear from the surface of the substrate, thereby preventing any further increase in the frictional force. No such force-limiting effect is present in prior art substrates, at least not until the frictional force is very high.

[0041] The second relevant mechanism is believed to be that as fibres are caused to protrude from the substrate they can be felt by the skin with which the substrate is in contact, and since these fibres, even if relatively stiff, are then free to bend, the sensation to the user is one of softness. However, the increased in perceived softness may result from other effects in addition to, or instead of, those just described, and no reliance is to be placed on the particular theoretical explanation just given.

[0042] Four substrates according to the invention (Examples 1 to 4) will now be described, together with a comparative example (Example 5) of a substrate not according to the invention.

[0043] Examples 1 to 4 were made from the same fibre composition, namely 50% viscose and 50% polypropylene, all the fibres having a length of 4cm and a diameter equivalent to 1.7dtex. Examples 1 to 4 were all made in the same way. The only difference between them was basis weight of the substrate, which was as follows (the basis weight of Example 5 also being given for completeness):

Ex 1	60.7 g/m ²
Ex 2	62.8 g/m ²
Ex 3	55.8 g/m ²
Ex 4	59.2 g/m ²
Ex 5	63.2 g/m ²

[0044] It should be noted that Example 5 used the same fibre composition and fibre diameter as Examples 1-4.

[0045] The apparatus used in Examples 1 to 4 had an apertured belt with 25 apertures/cm arranged in a square grid. The line travelled at about 50 m/min. 13 orifice arrays were used, each having about 1666 orifices per metre of their length, with each orifice being about 100 micrometers in diameter. Example 5 was made using a belt which was not apertured. In all of Examples 1-5 the jets were directed perpendicularly to the fibrous web. Other process conditions for Examples 1 to 4 and comparative Example 5 are set out in the following Table 1.

TABLE 1

	Ex.1-4	Ex.5
Combined flow rate through all jet arrays (m ³ /hr/array/m)	50	50
Pressure (bar)	45-85	40-90
Jet velocity (m/sec)	60-90	60-90
Energy flux per jet (bars.m/sec)	3000-7500	2000-8000
Total pressure across all arrays (bars)	620	970
Total power input from all arrays (kW/m web width)	62	107
Total energy input from all arrays (kWh/kg web)	0.34	0.59

[0046] The ranges given for pressure and jet velocity represent the fact that these increase for one array to the next, as considered in the direction of travel of the web. It must be understood that all values are approximate, and are given as accurately as they are known to the applicants.

[0047] Various properties of the substrate of Examples 1 to 5 were tested, and also a further comparative substrate, Example 6, which, like Examples 1-4, was a hydroentangled substrate, but, unlike comparative Example 5, was apertured. Example 6 was produced using more energy than for Examples 1 to 4. Example 6 had a basis weight of 53.9

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gm⁻². The results of these tests are set out in Table 2. The way in which the various tests were carried out is described later in this specification.

TABLE 2

	Ex 1	Ex 2	Ex 3	Ex 4	Ex 5	Ex 6
% apertures open	[10-20]	[10-20]	[10-20]	[10-20]	n/a	100
Caliper (mm)	0.8	0.79	0.79	0.79	0.56	0.68
Web density						
gcm ⁻³	0.075	0.079	0.069	0.075	0.113	0.079
Tensile strength (CD)						
at break (N)	6.96	6.1	6.5	7.7	9.0	7.0
Tensile strength (MD)						
at break (N)	35.7	27.2	25.4	30.2	49.3	64.0
Toughness (CD) (Nm)		0.4			0.6	0.6
Toughness (MD) (Nm)		0.9			1.5	1.5
Modulus (CD)(N% elongation)		0.016			0.032	0.05
Modulus (MD)(N% elongation)		0.8			1.6	7.5
%Elongation at break (CD)	114.7	140.4	123.9	120.7	156	150
%Elongation at break (MD)	32.1	31	29.7	29.2	33	25.1
Static friction (CD)(N)		1.71			1.91	1.79
Static friction (MD)(N)		1.54			1.91	1.77
Dynamic friction coefficient (CD)		0.018			0.023	0.021
Dynamic friction coefficient (MD)		0.017			0.024	0.023
Air permeability (l/m ² /sec)		1000			7490	12400
Absorption capacity (gH ₂ O/gweb)		9.07	9.57	9.35	8.5	

[0048] The values given for modulus are those for initial modulus, as is also the case in Table 2 below.

[0049] From the above it will be particularly noted that in the invention the initial CD modulus is very low, and that even though there are relatively few apertures the density is also low.

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[0050] A further set of tests were carried out, repeating some of those which are the subject of Table 2, on Examples 2, 5 and 6, after they had been subjected to a rubbing action. This action was intended to simulate what the substrate experiences when in use in, for example, a wet wipe. The results are set out in Table 3.

TABLE 3

	Ex.2	Ex.5	Ex.6
% apertures open	<5	n/a	100
Caliper (mm)	1.05	0.66	0.68
Tensile strength (CD) at break (N)	4.5	10	6.8
Tensile strength (MD) at break (N)	18	47	63
Toughness (CD) (Nm)	0.3	0.6	0.7
Toughness (MD) (Nm)	0.5	1.3	1.6
Modulus (CD)(N% elongation)	0.008	0.024	0.093
Modulus (MD)(N% elongation)	0.89	1.6	6.7

[0051] As can be seen from the above, rubbing substantially alters the properties of Example 2 (which is according to the invention), but has a much less marked effect on Examples 5 and 6 (which are not). In each case the alteration in Example 2 is consistent with its being significantly weaker than Examples 5 and 6, the effect of rubbing being to cause the substrate to break up to an extent sufficient to produce the desired sensation to the user, but not such as to cause complete disintegration (which is undesired).

[0052] In particular, attention is drawn to the fact that in Example 2 the caliper is significantly increased and the toughness is significantly decreased, both of which reflect the partial break-up of the substrate.

[0053] A comparison of Figures 2 and 3 shows the extent to which rubbing causes a substrate according to the invention (Example 2) to being to break up.

[0054] A comparison of Figure 4 (Example 2 after rubbing) and Figure 5 (Example 6 after rubbing) shows how much lower is the density of the substrate of the present invention, at least after rubbing, and the presence of numerous protruding fibres.

[0055] Various modifications may be made to the invention as described above, and some of these will now be mentioned:

(a) Instead of using orifice arrays for hydroentangling in which the orifices are uniformly spaced at, say, 1mm centres along the entire effective length of the array, each array may have a region of, say, 1cm, in which there are orifices spaced at 100 micrometer centres, alternately with regions of, say, 0.5cm in which there are no orifices. The effect of this is to produce a substrate which has alternating strips of entangled fibres and non-entangled fibres. The former provide strength to the substrate, and the latter provide fibres which readily come loose from the substrate as a result of the friction of rubbing.

(b) The substrate could be multi-layer. One possible construction is a sandwich in which a central layer (which may be of hydroentangled fibres, but could be of virtually any material, e.g. a woven material, with external layers of fibres formed therein by hydroentangling according to the invention. This would have the cleaning properties and softness associated with the present invention, but the possibility of other properties, e.g. added strength, from the central layer. Another possible construction is one of two layers, of which at least one layer is formed by hydroentangling according to the invention.

[0056] There now follows a description of how certain tests referred to above are to be carried out.

[0057] The following tensile properties of the previous examples are evaluated with an Instron tester under the following conditions. All tests are conducted at laboratory conditions of 21 C and 65% relative humidity. The Instron gauge length is 10 cm. Elongation rate is 10cm/min or 100%/minute. The Instron "jaws" that secure the sample are flat and rubber coated.

[0058] Tensile strength, initial modulus and toughness in both the MD and CD directions are determined from 1" wide strips cut to 15 cm in length and fixed without slack but without tension on the Instron tester within jaws set at 10 cm distance. The energy input from the Instron machine to the sample is then plotted over time with the y axis indicating the force applied to the sample in Newtons and the x axis indicating the % elongation of the sample at the indicated elongation rate.

[0059] The tensile strength number is defined as the peak force from this force over elongation curve.

[0060] The initial modulus number is obtained from the graph produced from the same test. It is the initial slope of the force/elongation curve and is indicated as the y axis in units of Newtons/% elongation.

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[0061] The toughness is the number obtained also from this same test and results graph. Toughness is defined as the area under the entire curve indicated in Newtons by % elongation.

[0062] Substrate caliper is measured using standard EDANA non-woven industry methodology, reference method #30-4-89.

5 [0063] Entanglement frequency is evaluated from the following data produced on the Instron tester with strips of varying widths, as indicated.

Strip Width indication	Avg CD Strip Tensile Example#2	Strip Width (in)	Instron Gauge Length (in)	Elongation Rate (in/ minute)
w0	8.0	0.8	0	0.5
w1	8.2	0.3	1.5	5
w2	17.1	1.9	1.5	5

15 [0064] From example #2, the entanglement frequency is calculated from the following equation.

$$\text{entanglement frequency CD} = \frac{2(T2-T1)}{w1T2-w2T1}$$

20 [0065] From this, then the entanglement frequency for example #2 in the CD direction is calculated to be 11.7. Further details of the concept of entanglement frequency can be found in the above cited reference US-A-3485706.

Abrasion or Rubbing Test

25 [0066] In this test the substrate to be tested is rubbed against another fixed piece of the same substrate to be tested.
[0067] One standard piece of equipment appropriate to do this is a "Satra Martindale Abrasion Machine" as described in the Journal of Textile Instruments; Vol 33 No 9 Sept 1942. However, any machine or laboratory set up that provides the following conditions may be used.

30 [0068] The test substrate to be evaluated is clamped into a fixed, taught but not stretched position on a horizontal surface. The circular clamp holding this material in place is 5" in diameter. The rubbing test is then conducted on the area of the sample within this fixed circular clamp.

[0069] A second sample of the test material is then clamped taught but not stretched in a fixed position on a 1.75" diameter flat and solid ended cylinder. This second sample is then rubbed on the first sample for the purpose of the test. The vertical force, or absolute mass (weight) of the second sample against the first sample is 0.68 Kg.

35 [0070] The second sample with this weight is then rubbed against the first sample in a repeating pattern, known as a Lissajous pattern, that fully covers the test sample, as shown in Figure 7.

[0071] There are 10 cycles of this pattern for the test where one cycle is represented by the full pattern shown above. One cycle consists of 15 oscillations in one direction and a second 15 oscillations at right angles to the first set of oscillations. One full cycle of these 30 oscillations is done over 18 seconds.

40 [0072] The substrate within the 5" fixed ring is then considered the treated sample. For the purposes of the Instron testing of the rubbed or treated sample, this piece of substrate is then cut in the CD or MD direction into strips of the appropriate width and length for the Instron test.

[0073] For example, for the tensile, initial modulus and toughness tests on the Instron machine, this treated strip is cut to 1" wide by 15 cm long strips, one such strip being indicated by the rectangle S in Figure 7.

45 [0074] Figure 6a to 6e are curves of the type just mentioned derived from three different materials, as follows:

Figure 6a/6b: Substrate according to comparative Example 5

Figure 6c/dc: Substrate according to the invention (Example 2)

Figure 6e/6f: Substrate according to comparative Example 6.

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[0075] Figures 6a, 6c and 6e are for MD, and Figures 6b, 6d and 6f are for CD. The rotation "NORMAL" means before rubbing and "RUBBED" means after rubbing.

[0076] The ordinate in each the graphs is in N, and the abscissa is in % elongation of the sample. For the purposes of calculating the toughness in Nm, the % value is converted into m by %/100 x 0.1m, with 0.1m being the length of the strips being tested.

55 [0077] The roughness values obtained are as follows :

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	Normal (Nm)	Rubbed (Nm)
Figure 6a	1.5	1.3
6b	0.6	0.6
6c	0.9	0.5
6d	0.4	0.3
6e	1.6	1.6
6f	0.6	0.7

Claims

1. A non-woven fibrous substrate for use in a cleaning article, wherein the substrate has a major surface for rubbing on a surface to be cleaned, a low strength in at least one direction so as to have a toughness as defined herein of less than 0.6Nm, and comprises at least 20% of long fibres which are capable of protruding from the said major surface as a result of rubbing as defined herein whilst remaining attached to the substrate.
2. A substrate according to claim, 1, which, as made, has a toughness, as defined herein, of not more than 0.5 Nm in at least one direction.
3. A substrate according to any preceding claim, which, as made, has a toughness, as defined herein, of less than 1.2Nm in each of two mutually perpendicular directions.
4. A substrate according to any preceding claim, which has a toughness, as defined herein, after rubbing, as also defined herein, of less than 0.5 Nm in at least one direction.
5. A substrate according to any preceding claim, which has a toughness, as defined herein, after rubbing, as also defined herein, of less than 0.6 Nm in each of two mutually perpendicular directions.
6. A substrate according to any preceding claim, wherein rubbing, as herein defined, reduces the toughness, as also defined herein, by at least 15%.
7. A substrate according to any preceding claim, which, as made, has a tensile strength, as defined herein, of not more than 45N in any direction.
8. A substrate according to any preceding claim, which has a tensile strength, as defined herein, after rubbing, as also defined herein, of not more than 10N in at least one direction, and/or not more than 30N in each of two mutually perpendicular directions.
9. A substrate according to any preceding claim, wherein the said long fibres have a length of at least 2cm, preferably a length of from 2 to 6cm, and more preferably a length of at least 3cm, most preferably from 3 to 5cm.
10. A substrate according any preceding claim, wherein the said long fibres have a tensile strength of at least 2.5cN, preferably at least 5cN.
11. A substrate according to any preceding claim, wherein said major surface is textured.
12. A substrate according to claim 11, wherein said texture is in the form of a grid of depressions and ridges, of which not more 80%, preferably not more than 50%, of the depressions form apertures open to the opposite face of the substrate.
13. A substrate according to claim 11, wherein there are from 12-30 depressions/cm².
14. A wet wipe which comprises a substrate according to any preceding claim, and a cleaning liquid carried thereby.
15. A wet wipe according to claim 14, wherein the substrate has density, when dry of not more than 0.1 g/cm³.

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16. A wet wipe according to claim 14 or 15, wherein the cleansing liquid is aqueous, or has an aqueous continuous phase, and wherein the said long fibres comprises fibres which are hydrophobic.
- 5 17. A wet wipe according to claim 16, wherein the said hydrophobic fibres are of polyethylene terephthalate or polypropylene, or have an external layer thereof.
18. A wet wipe according to claim 16 or 17, wherein the cleaning liquid is an oil or has an oily continuous phase, and wherein the said long fibres comprise fibres which are hydrophilic.
- 10 19. A wet wipe according to claim 14, wherein the cleaning liquid is a silicone emulsion.
20. A wet wipe according to claim 18, wherein the said hydrophilic fibres are of viscose, cotton or flax.
- 15 21. A wet wipe according to any one of claims 14 to 20, wherein the said long fibres comprise a mixture of hydrophobic and hydrophilic fibres.
22. A wet wipe according to any of claims 14 to 21, wherein all or substantially all the fibres of the substrate are long fibres.
- 20 23. A method of producing a non-woven, fibrous substrate for use in a cleaning article, and having a major surface for rubbing on a surface to be cleaned, wherein a fibrous layer comprising fibres of which at least 20% are long fibres, is subjected to hydroentangling by means of jets of liquid which apply to the fibrous layer a force energy of not more than 0.8 kWh/kg of fibrous layer material, sufficient to produce a coherent substrate but low enough for at least some of said long fibres to be capable of protruding from said major surface as a result of rubbing as defined herein whilst remaining attached to the substrate.
- 25 24. A method according to claim 23, wherein the jets of liquid apply to the fibrous layer an energy of not more than 0.5 kWh/kg.
- 30 25. A method according to claim 24, wherein the said energy is at least 0.0005 kWh/kg.
26. A method according to any one of claims 23 to 25, wherein the jets of liquid are directed at the fibrous layer while the layer is travelling in a first direction at a speed of at least 25 m/min, preferably at least 40 m/min.
- 35 27. A method according to claim 26, wherein the jets of liquid are directed at the fibrous layer at an angle which, as viewed in said first direction and/or as viewed perpendicular to said first direction, departs from the normal to the fibrous layer by a predetermined amount, preferably up to 45°.
- 40 28. A method according to any one of claims 23 to 27, wherein the fibrous layer is positioned on a carrier surface having apertures therein, preferably from 12 to 30 apertures/cm².

Patentansprüche

- 45 1. Ein faseriges Vlies-Substrat zur Verwendung in einem Reinigungsartikel, wobei das Substrat eine Hauptoberfläche zum Reiben an einer zu reinigenden Oberfläche, eine niedrige Festigkeit in mindestens einer Richtung aufweist, sodaß es eine Zähigkeit, gemäß der Definition hierin, von weniger als 0,6 Nm hat und mindestens zu 20 % lange Fasern enthält, die imstande sind, als eine Folge des Reibens, gemäß der Definition hierin, von der genannten Hauptoberfläche vorzuragen, während sie an das Substrat gebunden bleiben.
- 50 2. Ein Substrat nach Anspruch 1, das, wie hergestellt, in mindestens einer Richtung eine Zähigkeit, gemäß der Definition hierin, von nicht mehr als 0,5 Nm aufweist.
3. Ein Substrat nach einem der vorhergehenden Ansprüche, das, wie hergestellt, in jeder von zwei zueinander senkrechten Richtungen eine Zähigkeit, gemäß der Definition hierin, von weniger als 1,2 Nm aufweist.
- 55 4. Ein Substrat nach einem der vorhergehenden Ansprüche, das in mindestens einer Richtung eine Zähigkeit, gemäß der Definition hierin, nach dem Reiben, ebenfalls gemäß der Definition hierin, von weniger als 0,5 Nm aufweist.

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5. Ein Substrat nach einem der vorhergehenden Ansprüche, das in jeder von zwei zueinander senkrechten Richtungen eine Zähigkeit, gemäß der Definition hierin, nach dem Reiben, ebenfalls gemäß der Definition hierin, von weniger als 0,6 Nm aufweist.
- 5 6. Ein Substrat nach einem der vorhergehenden Ansprüche, worin das Reiben, gemäß der Definition hierin, die Zähigkeit, ebenfalls gemäß der Definition hierin, um mindestens 15 % herabsetzt.
7. Ein Substrat nach einem der vorhergehenden Ansprüche, das, wie hergestellt, in jeder Richtung eine Reißfestigkeit, gemäß der Definition hierin, von nicht mehr als 45 N aufweist.
- 10 8. Ein Substrat nach einem der vorhergehenden Ansprüche, das eine Reißfestigkeit, gemäß der Definition hierin, nach dem Reiben, ebenfalls gemäß der Definition hierin, von nicht mehr als 10 N in mindestens einer Richtung und/oder von nicht mehr als 30 N in jeder von zwei zueinander senkrechten Richtungen aufweist.
- 15 9. Ein Substrat nach einem der vorhergehenden Ansprüche, worin die genannten langen Fasern eine Länge von mindestens 2 cm, vorzugsweise eine Länge von 2 bis 6 cm, und bevorzugter eine Länge von mindestens 3 cm, am bevorzugtesten von 3 bis 5 cm, aufweisen.
- 20 10. Ein Substrat nach einem der vorhergehenden Ansprüche, worin die genannten langen Fasern eine Reißfestigkeit von mindestens 2,5 cN, vorzugsweise von mindestens 5 cN, aufweisen.
11. Ein Substrat nach einem der vorhergehenden Ansprüche, worin die genannte Hauptoberfläche texturiert ist.
- 25 12. Ein Substrat nach Anspruch 11, worin die genannte Textur in der Form eines Gitters mit Vertiefungen und Rippen vorliegt, wobei nicht mehr als 80 %, vorzugsweise nicht mehr als 50 %, der Vertiefungen Öffnungen bilden, die zu der gegenüberliegenden Seite des Substrats offen sind.
13. Ein Substrat nach Anspruch 11, worin 12-30 Vertiefungen/cm² vorliegen.
- 30 14. Ein Naßwischtuch, das ein Substrat nach irgendeinem vorhergehenden Anspruch und eine von demselben getragene Reinigungsflüssigkeit umfaßt.
15. Ein Naßwischtuch nach Anspruch 14, worin das Substrat im trockenen Zustand eine Dichte von nicht mehr als 0,1 g/cm³ aufweist.
- 35 16. Ein Naßwischtuch nach Anspruch 14 oder 15, worin die Reinigungsflüssigkeit wässrig ist oder eine wässrige kontinuierliche Phase hat und worin die genannten langen Fasern Fasern umfassen, die hydrophob sind.
- 40 17. Ein Naßwischtuch nach Anspruch 16, worin die genannten hydrophoben Fasern aus Polyethylenterephthalat oder Polypropylen bestehen oder eine äußere Schichte daraus aufweisen.
18. Ein Naßwischtuch nach Anspruch 16 oder 17, worin die Reinigungsflüssigkeit ein Öl ist oder eine ölige kontinuierliche Phase hat und worin die genannten langen Fasern Fasern umfassen, die hydrophil sind.
- 45 19. Ein Naßwischtuch nach Anspruch 14, worin die Reinigungsflüssigkeit eine Silikon-Emulsion ist.
20. Ein Naßwischtuch nach Anspruch 18, worin die genannten hydrophilen Fasern aus Viskose, Baumwolle oder Leinen bestehen.
- 50 21. Ein Naßwischtuch nach einem der Ansprüche 14 bis 20, worin die genannten langen Fasern eine Mischung von hydrophoben und hydrophilen Fasern umfassen.
22. Ein Naßwischtuch nach einem der Ansprüche 14 bis 21, worin alle oder im wesentlichen alle Fasern des Substrats lange Fasern sind.
- 55 23. Ein Verfahren zur Herstellung eines faserigen Vlies-Substrats zur Verwendung in einem Reinigungsartikel und das eine Hauptoberfläche zum Reiben an einer zu reinigenden Oberfläche aufweist, bei welchem Verfahren eine faserige Schichte, die Fasern umfaßt, von denen mindestens 20 % lange Fasern sind, einer Hydroverfilzung mit

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Hilfe von Flüssigkeitsstrahlen ausgesetzt wird, die auf die faserige Schichte eine mechanische Energie von nicht mehr als 0,8 kWh/kg Material der faserigen Schichte aufbringen, die ausreicht, um ein kohärentes Substrat zu bilden, jedoch niedrig genug ist, damit mindestens einige der genannten langen Fasern imstande sind, als eine Folge des Reibens, gemäß der Definition hierin, von der genannten Hauptoberfläche vorzuragen, während sie an das Substrat gebunden bleiben.

24. Ein Verfahren nach Anspruch 23, worin die Flüssigkeitsstrahlen auf die faserige Schichte eine mechanische Energie von nicht mehr als 0,5 kWh/kg aufbringen.

25. Ein Verfahren nach Anspruch 24, worin die genannte mechanische Energie mindestens 0,0005 kWh/kg beträgt.

26. Ein Verfahren nach einem der Ansprüche 23 bis 25, bei welchem die Flüssigkeitsstrahlen gegen die faserige Schichte gerichtet werden, während die Schichte sich in einer ersten Richtung mit einer Geschwindigkeit von mindestens 25 m/min, vorzugsweise von mindestens 40 m/min, bewegt.

27. Ein Verfahren nach Anspruch 26, worin die Flüssigkeitsstrahlen gegen die faserige Schichte in einem Winkel gerichtet werden, der bei Betrachtung in der genannten ersten Richtung und/oder bei Betrachtung senkrecht zu der genannten ersten Richtung, von der Normalen zu der faserigen Schichte um einen vorherbestimmten Betrag, vorzugsweise bis zu 45°, abweicht.

28. Ein Verfahren nach einem der Ansprüche 23 bis 27, worin die faserige Schichte an einer Trägeroberfläche angeordnet ist, die Öffnungen darin, vorzugsweise 12 bis 30 Öffnungen/cm², aufweist.

Revendications

1. Substrat fibreux non tissé pour l'utilisation dans un article de nettoyage, dans lequel le substrat a une surface principale pour frotter une surface devant être nettoyée, une faible résistance mécanique dans au moins une direction de manière à avoir une ténacité telle que définie dans la présente description inférieure à 0,6 Nm, et comprend au moins 20% de fibres longues qui sont capables de faire saillie de ladite surface principale par suite du frottement comme défini dans la présente description, tout en restant attachées au substrat.

2. Substrat selon la revendication 1 qui, tel que réalisé, a une ténacité, telle que définie dans la présente description, qui n'est pas supérieure à 0,5 Nm dans au moins une direction.

3. Substrat selon l'une quelconque des revendications précédentes, qui, tel que réalisé, a une ténacité, telle que définie dans la présente description, inférieure à 1,2 Nm dans chacune de deux directions mutuellement perpendiculaires.

4. Substrat selon l'une quelconque des revendications précédentes, qui a une ténacité, telle que définie dans la présente description, après frottement, tel que défini aussi dans la présente description, de moins de 0,5 Nm dans au moins une direction.

5. Substrat selon l'une quelconque des revendications précédentes, qui a une ténacité, telle que définie dans la présente description, après frottement, tel que défini aussi dans la présente description, de moins de 0,6 Nm dans chacune de deux directions mutuellement perpendiculaires.

6. Substrat selon l'une quelconque des revendications précédentes, dans lequel le frottement, tel que défini dans la présente description, réduit la ténacité, telle que définie aussi dans la présente description, d'au moins 15%.

7. Substrat selon l'une quelconque des revendications précédentes, qui, tel que réalisé, a une résistance à la traction, telle que définie dans la présente description, qui n'est pas supérieure à 45N dans n'importe quelle direction.

8. Substrat selon l'une quelconque des revendications précédentes, qui a une résistance à la traction, telle que définie dans la présente description, après frottement, tel que défini aussi dans la présente description, qui n'est pas supérieure à 10N dans au moins une direction, et/ou pas supérieure à 30N dans chacune de deux directions mutuellement perpendiculaires.

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9. Substrat selon l'une quelconque des revendications précédentes, dans lequel lesdites fibres longues ont une longueur d'au moins 2 cm, de préférence une longueur de 2 à 6 cm, et mieux encore une longueur d'au moins 3 cm, bien mieux encore de 3 à 5 cm.
- 5 10. Substrat selon l'une quelconque des revendications précédentes, dans lequel lesdites fibres longues ont une résistance à la traction d'au moins 2,5 cN, de préférence d'au moins 5 cN.
11. Substrat selon l'une quelconque des revendications précédentes, dans lequel ladite surface principale est texturée.
- 10 12. Substrat selon la revendication 11, dans lequel ladite texture est sous la forme d'une grille de dépressions et de crêtes, pour laquelle pas plus de 80%, de préférence pas plus de 50%, des dépressions forment des jours ouverts sur la face opposée du substrat.
13. Substrat selon la revendication 11, dans lequel il existe de 12 à 30 dépressions/cm².
- 15 14. Lingette imbibée qui comprend un substrat selon l'une quelconque des revendications précédentes, et un liquide de nettoyage porté par celle-ci.
- 20 15. Lingette imbibée selon la revendication 14, dans laquelle le substrat a une masse volumique, quand il est sec, qui n'est pas supérieure à 0,1 g/cm³.
16. Lingette imbibée selon la revendication 14 ou 15, dans laquelle le liquide de nettoyage est aqueux, ou a une phase continue aqueuse, et dans laquelle lesdites fibres longues comprennent des fibres qui sont hydrophobes.
- 25 17. Lingette imbibée selon la revendication 16, dans laquelle lesdites fibres hydrophobes sont en poly(téréphtalate d'éthylène) ou en polypropylène, ou ont une couche externe constituée de ces composants.
18. Lingette imbibée selon la revendication 16 ou 17, dans laquelle le liquide de nettoyage est une huile ou a une phase continue huileuse, et dans laquelle lesdites fibres longues comprennent des fibres qui sont hydrophiles.
- 30 19. Lingette imbibée selon la revendication 14, dans laquelle le liquide de nettoyage est une émulsion de silicone.
20. Lingette imbibée selon la revendication 18, dans laquelle lesdites fibres hydrophiles sont en viscose, coton ou lin.
- 35 21. Lingette imbibée selon l'une quelconque des revendications 14 à 20, dans laquelle lesdites fibres longues comprennent un mélange de fibres hydrophobes et hydrophiles.
22. Lingette imbibée selon l'une quelconque des revendications 14 à 21, dans laquelle toutes ou sensiblement toutes les fibres du substrat sont des fibres longues.
- 40 23. Procédé de fabrication d'un substrat fibreux non tissé pour l'utilisation dans un article de nettoyage, et ayant une surface principale pour frotter une surface devant être nettoyée, dans lequel on soumet une couche fibreuse comprenant des fibres dont au moins 20% sont des fibres longues, à un emmêlement par l'eau au moyen de jets de liquide qui appliquent à la couche fibreuse une énergie de force qui n'est pas supérieure à 0,8 kWh/kg du matériau en couche fibreuse, suffisante pour produire un substrat cohérent mais suffisamment faible pour qu'au moins quelques-unes desdites fibres longues soient capables de faire saillie de ladite surface principale par suite du frottement comme défini dans la présente description, tout en restant attachées au substrat.
- 45 24. Procédé selon la revendication 23, dans lequel les jets de liquide appliquent à la couche fibreuse une énergie qui n'est pas supérieure à 0,5 kWh/kg.
- 50 25. Procédé selon la revendication 24, dans lequel ladite énergie est d'au moins 0,0005 kWh/kg.
26. Procédé selon l'une quelconque des revendications 23 à 25, dans lequel les jets de liquide sont dirigés sur la couche fibreuse pendant que la couche se déplace dans une première direction à une vitesse d'au moins 25 m/min, de préférence d'au moins 40 m/min.
- 55 27. Procédé selon la revendication 26, dans lequel les jets de liquide sont dirigés sur la couche fibreuse selon un

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angle qui, tel que vu dans une première direction et/ou vu perpendiculairement à ladite première direction, dévie de la normale à la couche fibreuse d'une quantité prédéterminée, de préférence jusqu'à 45°.

28. Procédé selon l'une quelconque des revendications 23 à 27, dans lequel la couche fibreuse est placée sur une surface support munie d'ouvertures, de préférence de 12 à 30 ouvertures/cm².

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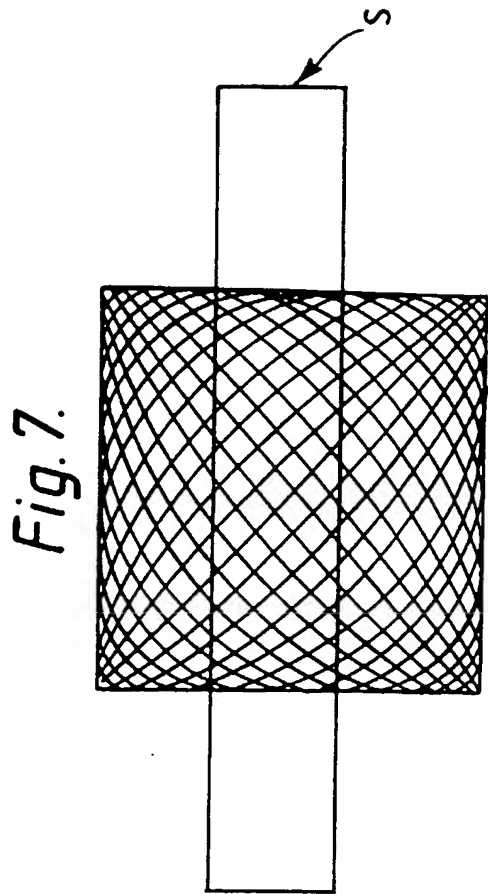
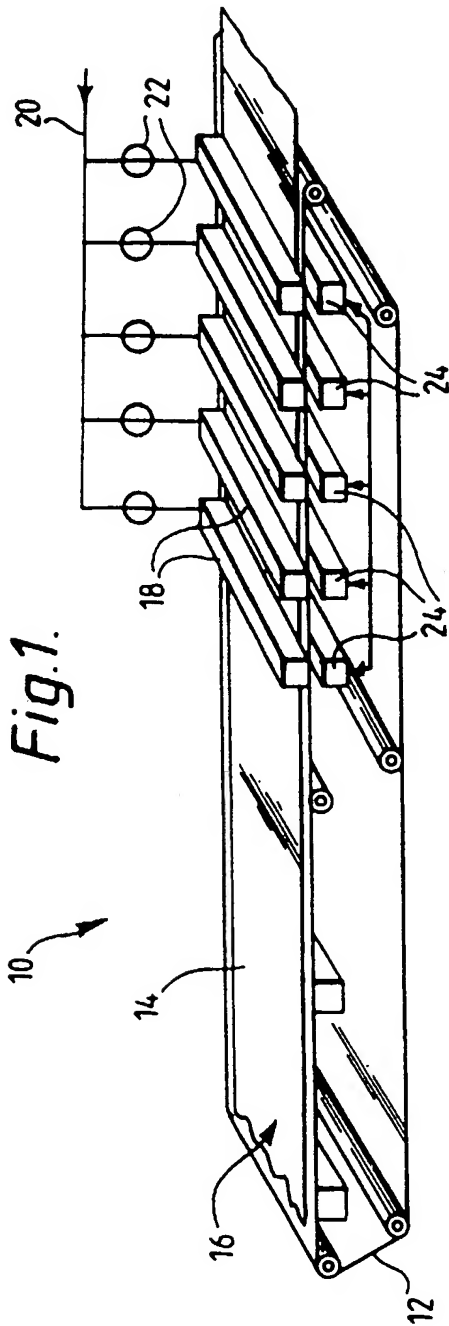
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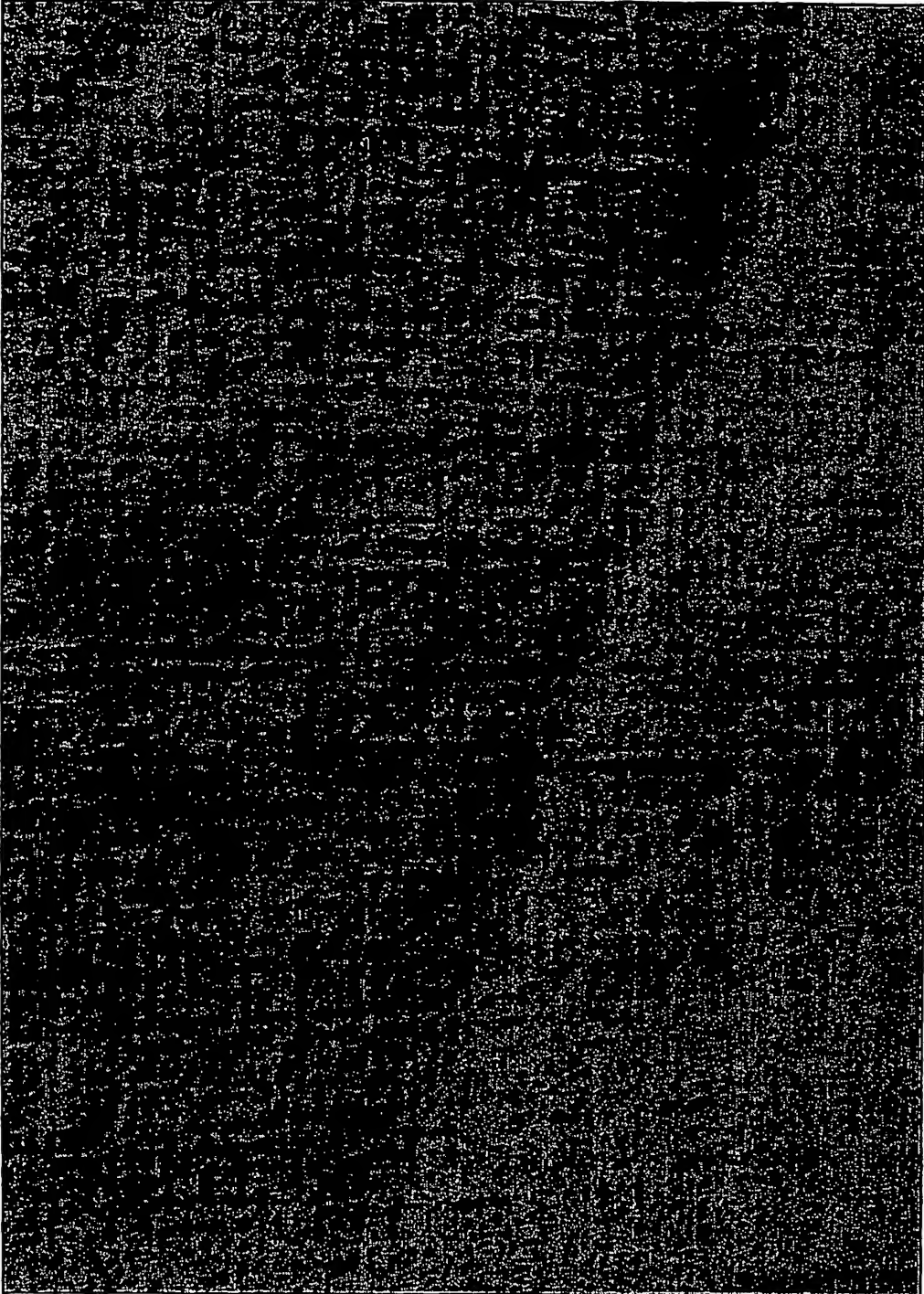
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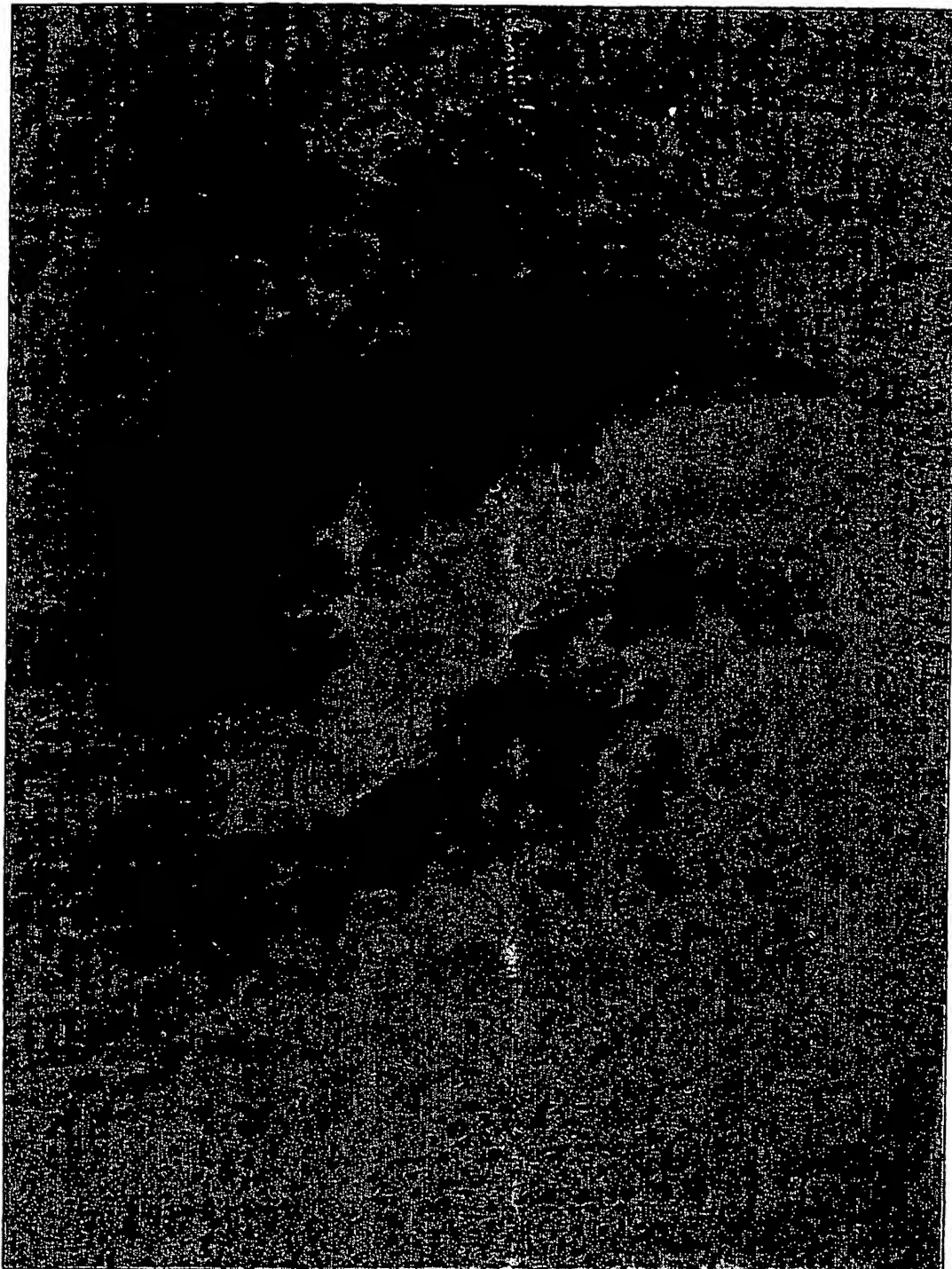
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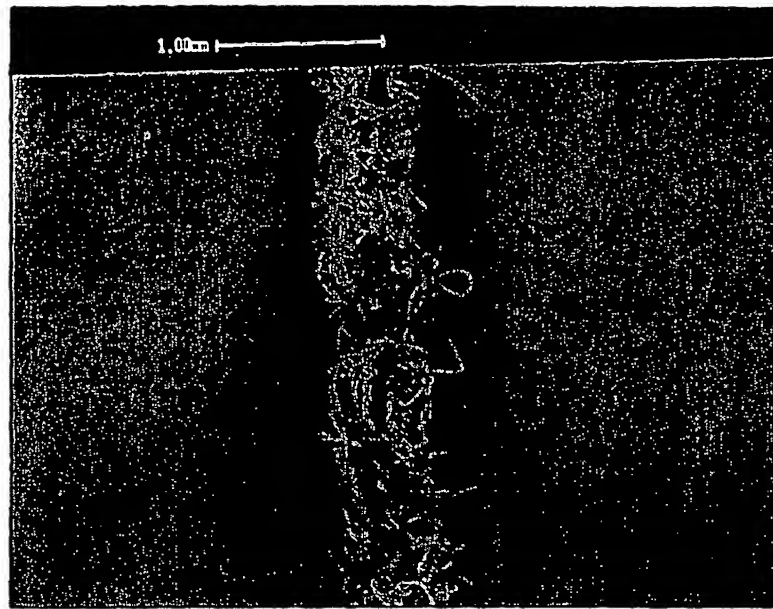
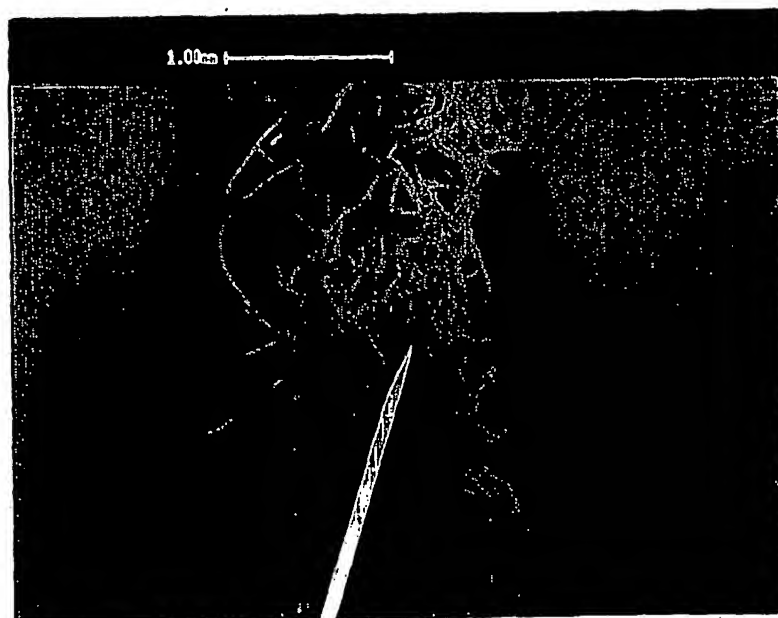
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Fig. 2**BEST AVAILABLE COPY**

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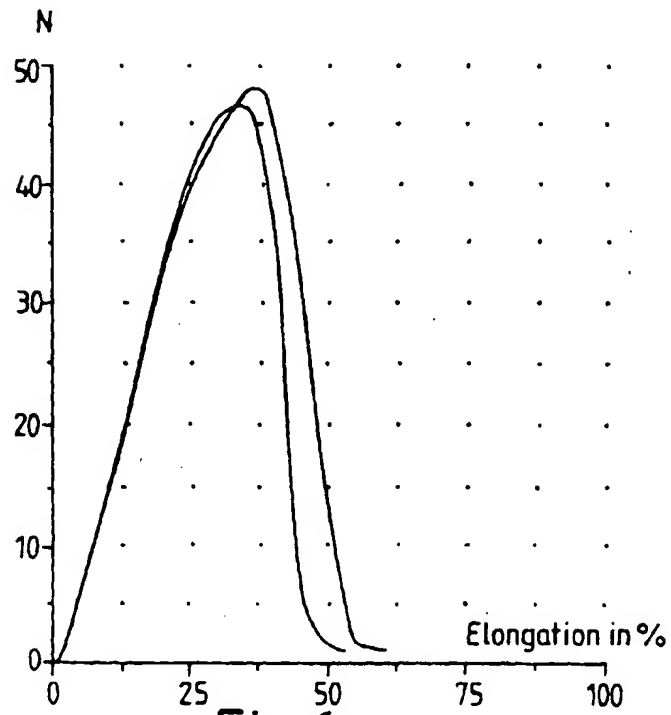
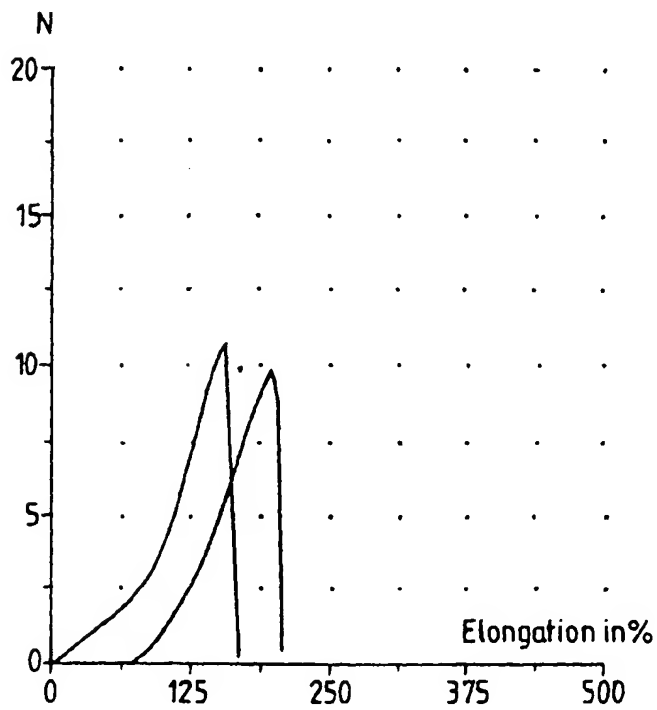
Fig. 3**BEST AVAILABLE COPY**

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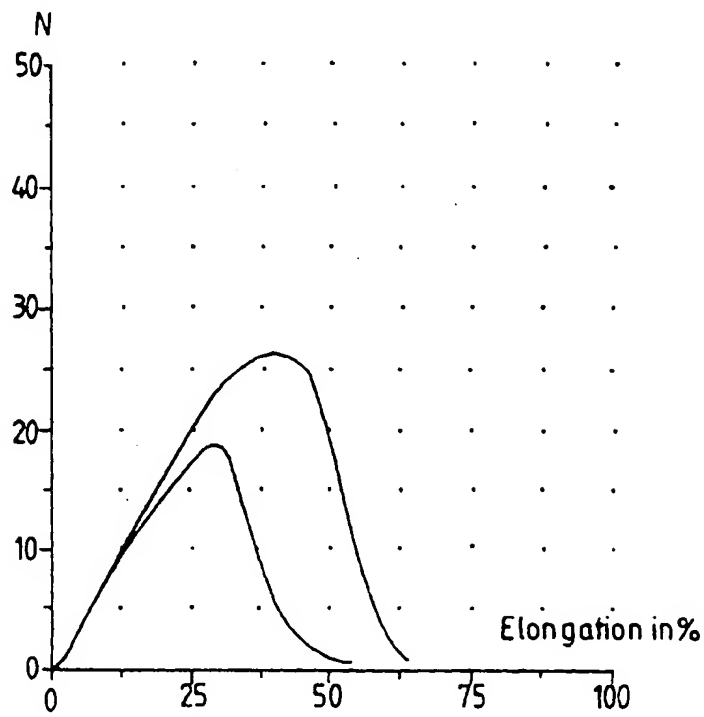
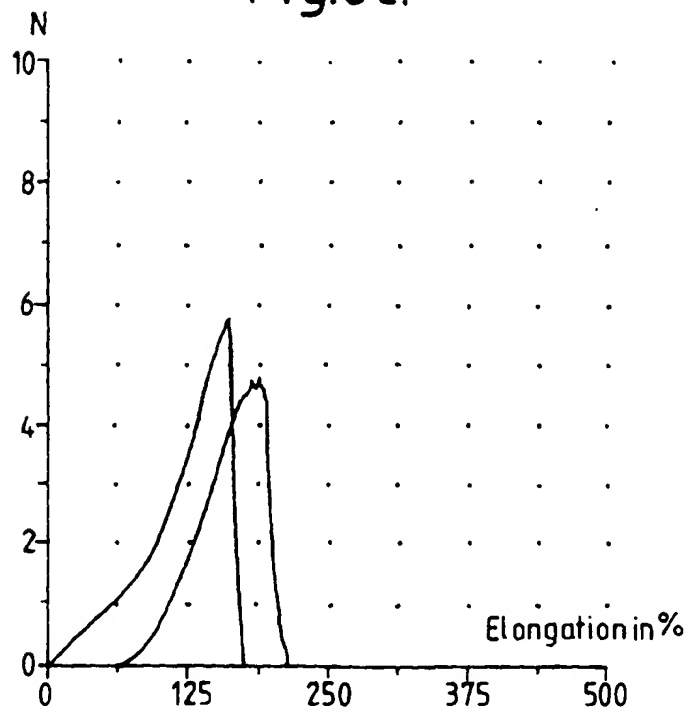
Fig. 4*Fig. 5*

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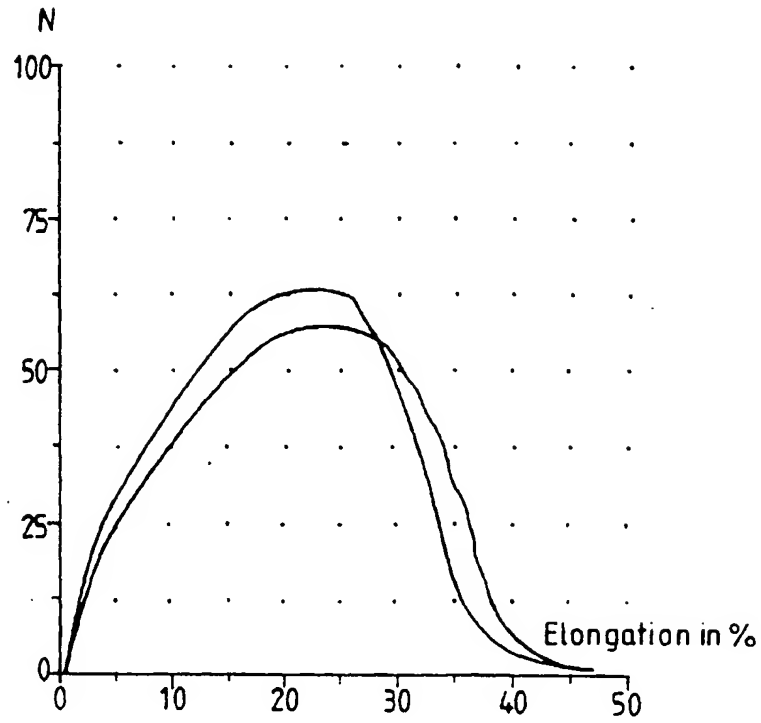
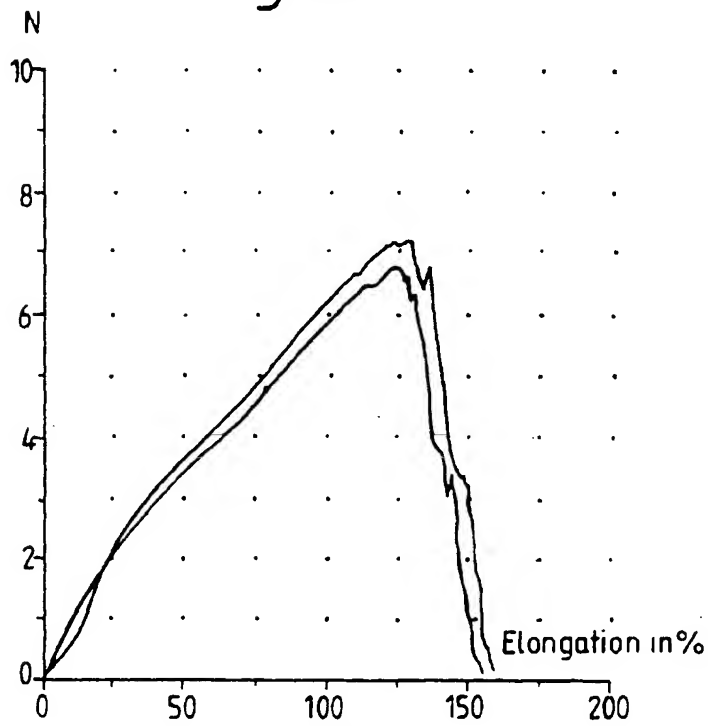
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*Fig. 6a.**Fig. 6b.*

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*Fig. 6c.**Fig. 6d.*

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*Fig. 6e.**Fig. 6f.*